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THE DETERMINATION OF MOISTURE IN COKE

BY

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CONTENTS.

	Page.
Introduction	5
Review of the literature	6
Effect of dry-air circulation and duration of heating	7
Effect of temperature	8
Direct determination of moisture in lump coke	9
Determination of moisture by drying lump samples on hot plate	11
Summary	12
Publications on the composition of coal	13

TABLES.

	Page.
TABLE 1. Results of determining moisture at 105° C. in 60-mesh coke, with and without dry-air circulation	8
2. Results of determination of moisture in 60-mesh coke at various temperatures	8
3. Percentage of moisture in air-dry coke at various stages in the preparation of the 60-mesh laboratory sample	10
4. Results obtained by heating 4-mesh coke to various temperatures	10
5. Results obtained by heating 1-inch lumps of coke to constant weight on a hot plate at various temperatures	11

THE DETERMINATION OF MOISTURE IN COKE.

ARNO C. FIELDNER and WALTER A. SELVIG.

INTRODUCTION.

The experiments described in this paper were undertaken, in the course of fuel investigations made by the Bureau of Mines, with the purpose of ascertaining the most rapid and simple method for determining the moisture in coke with sufficient accuracy for use in specifications for the purchase of coke by the Government, by industrial concerns, or by private individuals.

For the determination of moisture in coal, the committee on coal analysis of the American Society for Testing Materials and the American Chemical Society[a] recommend heating the pulverized sample (60-mesh) for one hour at 104° to 110° C. in an oven through which is passed a rapid current of air dried by passing through concentrated sulphuric acid. Under the same conditions 20-mesh samples must be heated 1½ hours.

In order to obtain comparable results, the method specified must be strictly followed, especially with coals normally having a high moisture content. Experiments have shown that such coals will easily give off 1 per cent more moisture in an atmosphere of circulating dry air than in an atmosphere of more or less stagnant air in which there is an appreciable partial pressure of water vapor. It is also essential, in order to obtain the correct moisture content, to pulverize the coal to a fineness of at least 10-mesh before the final moisture determination. The best practice is to air-dry the coal at ½ or ¼ inch size and then to pulverize it for the final determination of residual moisture, thus determining the water in two stages.

Undoubtedly the above method for the determination of moisture in coal gives results of the same degree of accuracy when applied to coke. However, owing to the much greater stability of coke as compared to coal, it is believed that a simpler method gives accurate results.

Coal is a complex mixture of extremely sensitive organic compounds containing a considerable proportion of hydrogen and oxygen. These compounds are easily altered by heating and by oxidation. On the other hand, coke is the residual product obtained from heating coal to a high temperature, and consists chiefly of carbon and inorganic ash-forming constituents. The hydrogen and oxygen content is less than 1 per cent, and the carbon is fused, so that the great number of very finely divided particles of the original coal substance, which afforded a vast surface system for the condensation of moisture films, are united.

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[a] Tentative methods for laboratory sampling and analysis of coal: Year Book, Am. Soc. Test. Mat., 1915, pp. 600-601.

It therefore seems probable that the circulation of dried air is unnecessary; that larger temperature limits than 104° to 110° C. are permissible; that no appreciable gain in weight due to absorption of oxygen takes place on heating longer than one hour; and, finally, that practically all the moisture should be removed on heating the unpulverized coke to constant weight. If the latter procedure is permissible, the ever-present danger of loss of moisture during pulverization will be eliminated and it will be possible to determine the moisture content of a shipment of coke with the same ease and simplicity as for iron ores.

REVIEW OF THE LITERATURE.

Comparatively few reports of experiments on the determination of moisture in coke have been published. Most textbooks recommend the same procedure as for coal. A few investigators have recognized that coke may be dried to constant weight in much the same manner as ore is dried.

Thörner[a] recommends that the gross sample of coke be pulverized and quartered to hazelnut size immediately after collection. A representative portion of 1,200 to 1,500 grams is placed in a galvanized-iron can, sealed moisture-tight, and shipped to the laboratory. At the laboratory a glass tube is inserted in a suitable opening in the can and pushed down until the end is near the bottom. The can is then weighed, placed in an air oven at 105 to 110° C., and heated to constant weight while a current of air is drawn through the tube. Usually 8 to 10 hours are required to remove all the moisture.

The 1912[b] report of the committee on standard methods for determining the constituents of foundry coke specifies that the gross sample of coke shall be crushed to ½-inch size, mixed, and quartered. One quarter shall be reserved for the determination of total moisture. This determination consists in drying to constant weight at 104 to 107° C.

Wagener[c] found that saturated lump coke containing 16 to 20 per cent water lost 4 per cent water while being pulverized and

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[a] Thörner, W., Beitrage zur bestimmung der Feuchtigkeit und Asche in Koks. Stahl und Eisen Bd. 8, Th. 2, 1888 S. 589-591.

[b] Report of the committee on standard methods for determining the constituents of foundry coke. Trans. Am. Foundrymen's Assoc., vol. 21, 1912, pp. 143-146.

[c] Wagener, A., Ueber den Wasser-und-Aschengehalt des Kokses. Ferrum, Bd. 10, 1913, pp. 321-336, 353-369.

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put through a sieve having 4-mm. meshes. Unsaturated coke containing 9 per cent water lost from 1 to 2 per cent water. To avoid this loss of moisture, Wagener recommended that the gross sample of coke be quickly broken down to fist size with a hammer, then mixed and quartered. Two opposite quarters weighing about 60 kilograms are weighed on a tared galvanized-iron pan, placed in a large oven and dried for 24 hours at a temperature of 150° C. The loss in weight is the total moisture. This dried sample is then pulverized and analyzed, it being assumed that moisture is reabsorbed from the atmosphere during the pulverizing process.

EFFECT OF DRY-AIR CIRCULATION AND DURATION OF HEATING.

The first series of tests by the authors of this paper were made to ascertain whether the circulation of dry air through the oven is necessary to remove the maximum amount of moisture. Twelve samples of different metallurgical cokes from various sources were prepared by crushing the air-dry coke to 60 mesh. Moisture determinations were made in duplicate by the standard method used by the Bureau of Mines for determining moisture in coal.[a] Samples weighing 1 gram were placed in ⅞-inch by 1¾-inch porcelain capsules and in sets of 12 heated for one hour at 105° C. in a constant-temperature oven. A current of air, dried by concentrated sulphuric acid, was passed through the oven and over the samples rapidly enough to change the total volume of air in the oven two to four times in one minute. At the end of the heating period the capsules were covered with closely fitting, flat aluminum covers and cooled in separate desiccators over concentrated sulphuric acid. Each capsule was rapidly weighed to the nearest 0.5 mg. immediately upon removal from the desiccator.

Duplicate determinations were also made in sets of 12 in the same oven without using dry-air circulation, and a fifth set of determinations was made in which the period of heating was extended to two hours, in order to ascertain whether any gain in weight through absorption of oxygen took place. Coal samples heated for two hours usually show less loss in weight than samples heated for one hour.

As shown in Table 1, no appreciable difference in moisture was obtained by circulating dry air through the oven. The difference for the 12 samples was only 0.005 per cent. Neither was there any appreciable change in weight on heating the samples for two hours.

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[a] Stanton, F. M., and Fieldner, A. C., Methods of analyzing coal and coke: Tech. Paper 8, Bureau of Mines, 1913, pp. 12-13.

Table 1.—*Results of determining moisture at 105° C. in 60-mesh coke, with and without dry-air circulation.*

Coke No.	Percentage of moisture.							Difference.	
	Heated 1 hour in current of dry air.			Heated 1 hour in stagnant air.			Heated 2 hours in stagnant air.	Columns 4 and 7.	Columns 7 and 8.
	Set 1.	Set 2.	Average.	Set 3.	Set 4.	Average.			
1	2	3	4	5	6	7	8	9	10
13651	0.15	0.16	0.16	0.15	0.16	0.16	0.19	0.00	+0.03
13672	.26	.27	.27	.26	.30	.28	.33	+.01	+ .05
13707	.20	.17	.19	.16	.21	.19	.21	.00	+ .02
13743	.63	.68	.66	.60	.67	.64	.71	−.02	+ .07
13784	.07	.10	.09	.07	.11	.09	.06	.00	− .03
13829	.12	.13	.13	.12	.10	.11	.09	−.02	− .02
13388	.11	.10	.11	.10	.10	.10	.10	−.01	.00
14172	.09	.13	.12	.12	.12	.12	.08	.00	− .04
14183	.33	.33	.33	.32	.33	.33	.30	.00	− .03
14730	.44	.48	.46	.47	.53	.50	.47	+.04	− .03
15276	.21	.22	.22	.21	.21	.22	.22	.00	.00
16933	.37	.36	.37	.30	.31	.31	.37	−.06	+ .06
Average								−.005	+.007

EFFECT OF TEMPERATURE.

The second series of tests were made to ascertain the effect of heating the 60-mesh coke at temperatures higher than 105° C. Three sets of determinations were made, namely, set No. 1 by the standard method at 105° C. as in the first series; set No. 2 by heating in stagnant air at 150° C.; and set No. 3 by heating in stagnant air at 200° C. Each set was heated for a period of 1 hour, then cooled over concentrated sulphuric acid, and weighed; then it was returned to the oven, heated for an additional 30 minutes, and again weighed. The results are given in Table 2.

Table 2.—*Results of determination of moisture in 60-mesh coke at various temperatures.*

Coke No.	Percentage of moisture.						Difference.	
	Heated in current of dry air at 105° C.		Heated in stagnant air at 150° C.		Heated in stagnant air at 200° C.		Columns 3 and 5.	Colums 3 and 7.
	1 hour.	1½ hours.	1 hour.	1½ hours.	1 hour.	1½ hours.		
1	2	3	4	5	6	7	8	9
13651	0.15	0.15	0.30	0.31	0.36	0.37	0.16	0.22
13672	.21	.22	.42	.42	.51	.53	.20	.31
13707	.20	.22	.28	.29	.32	.34	.07	.12
14172	.12	.13	.23	.23	.30	.30	.10	.17
13743	.66	.70	.84	.89	.95	.96	.19	.26
13784	.08	.09	.11	.11	.16	.18	.02	.09
14183	.31	.31	.49	.47	.62	.62	.16	.31
16933	.36	.39	.51	.51	.59	.59	.12	.20
14730	.54	.55	.65	.65	.79	.80	.10	.25
15276	.26	.26	.35	.36	.45	.47	.10	.21
13888	.11	.11	.15	.15	.23	.25	.04	.14
13829	.13	.13	.22	.22	.28	.28	.09	.15
Average							.11	.20

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For each of the three temperatures, 105°, 150°, and 200° C., the loss in weight for one hour was practically the same as that attained through additional heating for 30 minutes. There was, however, a slightly greater loss at the higher temperatures. The greatest difference between 105° and 150° C. was 0.20 per cent, and between 105° and 200° was 0.31 per cent. The average differences were 0.11 and 0.20 per cent respectively.

It is probable that the greater loss at the higher temperatures is due to the removal of residual traces of water and gases condensed in extremely thin films on the surfaces of the coke particles. Mack and Hulett [a] have called attention to these films in connection with the water in coal and have pointed out that such water would probably have no measurable vapor pressure at 100° C. but would gradually, as the temperature increased, develop sufficient vapor pressure to permit removal of additional moisture at higher temperature. However, the differences that were obtained on heating the coke powder between limits of 105° and 150° C. may be regarded as negligible; even the maximum difference of 0.31 per cent at 200° C. is within the unavoidable moisture variations likely to be incurred in preparing the gross sample for analysis.

DIRECT DETERMINATION OF MOISTURE IN LUMP COKE.

Experiments on the direct determination of moisture in 1-inch lumps and also in 4-mesh coke at various temperatures were made in the following manner:

A large sample of coke was taken from a pile that was wet from a recent rain and was crushed to 4-mesh size. This sample was then thoroughly mixed and divided into three representative portions, A1, A2, and A3, which were treated as described below.

Sample A1 was air-dried to constant weight at 30 to 35° C., then rapidly crushed to 10-mesh by passing it through crushing rolls. The 10-mesh product was quickly reduced on a riffle sampler to 400 grams, which was pulverized to 60-mesh in an air-tight porcelain Abbe ball mill containing flint pebbles. The 60-mesh sample was then reduced to 60 grams on a small riffle sampler and placed in a rubber-stoppered bottle. The residual moisture in this air-dried sample was determined at 105° C., according to the standard method previously described in this paper. The total moisture "as received" in sample A1 was computed as follows:

$$\text{Moisture ``as received''} = \text{Moisture in 60-mesh coke} \times \frac{(100-\text{air-drying loss})}{100} + \text{air-drying loss.}$$

In order to check any possible change of moisture content in the air-dry sample, while it was being pulverized and reduced from 4-mesh to 60-mesh, samples for moisture determination were taken at various stages of the sampling procedure as follows:

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[a] Mack, Edward, and Hulett, G. H.: The water content of coal, with some ideas on the genesis and nature of coal. Am. Jour. Sci., ser. 4, vol. 43, 1917, pp. 89-110.

TABLE 3.—*Percentage of moisture in air-dry coke at various stages in the preparation of the 60-mesh laboratory sample.*

Kind of sample.	Moisture.
	Per cent.
4-mesh sample taken immediately after air-drying; 341.5 grams dried to constant weight at 150° C.	0.32
10-mesh sample taken immediately after passing through rolls; 433.6 grams dried to constant weight at 150° C.	.23
60-mesh sample taken immediately after opening ball mill; 1 gram dried 1 hour at 105° C. in a current of dry air	.34
Final 60-gram laboratory sample; determination as above	.35

The maximum variation in the different samples was 0.12 per cent, which is of the same magnitude as the error in the moisture determination. Hence it may be assumed that the "as received" moisture as determined in sample A1 is the true moisture content of the coke and may be used as a standard of reference for judging the accuracy of the results that were obtained on samples B and C by shorter methods.

Sample A2 (at 4-mesh size) was dried to constant weight at a temperature of 105° C. in a Freas electrically heated oven without dry-air circulation; and sample A3 (at 4-mesh size) was similarly dried to constant weight in an ordinary gas-heated oven at a temperature of 150° C. The results obtained on the three samples are given in Table 4, together with the results on a second set of three samples, B1, B2, and B3, which were similarly prepared from another lot of coke.

TABLE 4.—*Results obtained by heating 4-mesh coke to various temperatures.*

Sample.	Fineness.	Temperature of drying.	Time required to reach constant weight.	Loss in weight.
		° C.	*Hours.*	*Per cent.*
Sample A1, 2,056 grams	4-mesh	30–35	120	10.1
Sample A1, 1 gram	60-mesh	105	1	0.35
Total moisture "as received"				10.4
Sample A2, 1,089 grams	4-mesh	95–105	2.5	10.5
Sample A3, 514 grams	4-mesh	150	2.0	10.5
Sample B1, 1,630 grams	4-mesh	30–35	72	9.7
Sample B1, 1 gram	60-mesh	105	1	0.38
Total moisture "as received"				10.0
Sample B2, 750.2 grams	4-mesh	95–105	2.5	9.7
Sample B3, 426.2 grams	4-mesh	150	2.0	9.7

This table shows that moisture can be determined in 4-mesh coke by drying to constant weight at temperatures between 105° and 150° C., with practically the same degree of accuracy as in the longer process of first air-drying at 4-mesh size and then pulverizing the coke to 60-mesh for final drying at 105° C. in circulating dry air.

DETERMINATION OF MOISTURE BY DRYING LUMP SAMPLES ON HOT PLATE.

The next step in the simplification of the method of determining moisture in coke was to ascertain whether it would be permissible to dry comparatively large samples (10 pounds or more) of 1 or 2 inch lumps to constant weight on an ordinary hot plate, coil of steam pipes, or similar device such as would be readily available at the point of sampling a shipment of coke. If such a procedure were possible the total moisture could be determined on the spot. The sample for chemical analysis could then be pulverized to 4-mesh size and quartered down to a 5-pound sample for shipment to the laboratory without special precautions for avoiding the almost inevitable loss of moisture.

A sample of approximately 50 pounds of lump coke was crushed to pass a 1-inch mesh sieve and thoroughly sprayed with water. It was then mixed for some time and allowed to stand several hours. The moist coke was then placed on a sampling cloth, again mixed, coned and divided into four parts, designated as C1, C2, C3, and C4. Each quarter was placed in a tarred galvanized-iron pan, 22 by 26 inches, and immediately weighed. The coke was spread to a depth of about 2 inches.

Sample C1 was placed in an air-drying oven and dried to constant weight at 30°–35° C. and then pulverized and reduced to a 60-mesh laboratory sample, as in the usual standard method.

The other three samples were dried to constant weight on ordinary gas-heated hot plates at temperatures of 120°, 150°, and 200° C., respectively. There was an air space of one-fourth inch between the bottom of the pans and the hot plate. The temperatures were determined by inserting thermometers in the bed of coke near the bottom of the pans. The results obtained are given in the following table:

TABLE 5.—*Results obtained by heating 1-inch lumps of coke to constant weight on a hot plate at various temperatures.*

Sample No.	Weight of sample.	Temperature of drying.	Time required to reach constant weight.	Loss in weight.
	Grams.	° *C.*	*Hours.*	*Per cent.*
C1	6,291	30–35	72	5.3
C1, 60-mesh	1	105	1	.38
Total moisture in C1				5.7
C2	5,105	120	2	5.9
C3	4,909	150	2	6.2
C4	5,923	200	1	5.9

The sample dried at 120° C. and the sample dried at 200° C. yielded 5.9 per cent moisture. These results were 0.2 per cent higher than that obtained with sample C1 which was air-dried before the moisture was determined by the standard method. The small differences in results with the various portions were probably due to unequal wetting rather than to the temperature of drying. Even the maximum difference is within the sampling error.

SUMMARY.

1. Investigation shows that the influence of temperature, time, humidity of drying atmosphere, and fineness of sample on the determination of moisture in coke may be varied over a considerable range without affecting the result appreciably.

2. Oven temperatures ranging from 105° to 200° C. produced a maximum variation in moisture of not exceeding 0.3 per cent.

3. Coke can be dried to "constant weight" without any gain in weight taking place.

4. The circulation of air dried by sulphuric acid, through the oven atmosphere, as specified for coal analysis, is unnecessary, there being no measurable difference of results between circulating perfectly dry air through the oven and using in the oven the natural circulation of air from the room.

5. Moisture can be determined quickly and with adequate accuracy ±0.5 per cent) by simply heating to constant weight a large sample of lump coke, in any convenient oven, or on a stove, hot plate, or steam coil at a temperature of 100° to 200° C.

Because of its simplicity and flexibility this method may be used advantageously at points when coke shipments are sampled.

PUBLICATIONS ON THE COMPOSITION OF COAL.

A limited supply of the following publications of the Bureau of Mines has been printed and is available for free distribution until the edition is exhausted. Requests for all publications can not be granted, and to insure equitable distribution applicants are requested to limit their selection to publications that may be of especial interest to them. Requests for publications should be addressed to the Director, Bureau of Mines.

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BULLETIN 85. Analyses of mine and car samples of coal collected in the fiscal years 1911 to 1913, by A. C. Fieldner, H. I. Smith, A. H. Fay, and Samuel Sanford. 1914. 444 pp., 2 figs.

BULLETIN 116. Methods of sampling delivered coal, and specifications for the purchase of coal for the Government, by G. S. Pope. 1916. 64 pp., 5 pls., 2 figs.

BULLETIN 119. Analyses of coals purchased by the Government during the fiscal years 1908–1915, by G. S. Pope. 1916. 118 pp.

TECHNICAL PAPER 2. The escape of gas from coal, by H. C. Porter and F. K. Ovitz. 1911. 14 pp., 1 fig.

TECHNICAL PAPER 5. Constituents of coal soluble in phenol, by J. C. W. Frazer and E. J. Hoffman. 1912. 20 pp., 1 pl.

TECHNICAL PAPER 8. Methods of analyzing coal and coke, by F. M. Stanton and A. C. Fieldner. 1913. 42 pp., 12 figs.

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BULLETIN 22. Analyses of coals in the United States, with descriptions of mine and field samples collected between July 1, 1904, and June 30, 1910, by M. W. Lord, with chapters by J. A. Holmes, F. M. Stanton, A. C. Fieldner, and Samuel Sanford. 1912. Part I, Analyses, pp. 1–321; Part II, Descriptions of samples, pp. 321–1129. 85 cents.

BULLETIN 29. The effect of oxygen in coal, by David White. 80 pp., 3 pls. 20 cents.

BULLETIN 38. The origin of coal, by David White and Reinhardt Thiessen, with a chapter on the formation of peat, by C. A. Davis. 1913. 390 pp., 54 pls. 80 cents.

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BULLETIN 63. Sampling coal deliveries and types of Government specifications for the purchase of coal, by G. S. Pope. 1913. 68 pp., 4 pls., 3 figs. 10 cents.

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